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## Liquid Crystals

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# Dimesogenic compounds consisting of cholesterol and azobenzene-based moieties: dependence of liquid crystal properties on spacer length and fluorination of the terminal chain

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The liquid crystalline (LC) properties of two series of non-symmetric dimesogenic compounds consisting of cholesterol and azobenzene-based moieties interconnected by  $\omega$ -oxyalkanoyl spacers of varying length are compared: one series (AOC-*n*) has an octyloxy chain attached to the azobenzene mesogen unit while the other (AOCF-*n*) has a perfluoroheptylmethoxy chain. In general, compounds bearing the fluorinated alkoxy chain exhibited LC properties over a much broader temperature range than those with the alkoxy chain. In addition, the AOC-*n* series exhibited the chiral smectic C (SmC\*), smectic A (SmA) and cholesteric (N\*) phases depending on the length of the central spacer, whereas the AOCF-*n* series favoured the formation of only the SmA phase with the N\* phase completely suppressed. Both series showed an odd–even dependence of the isotropization temperature on spacer length.

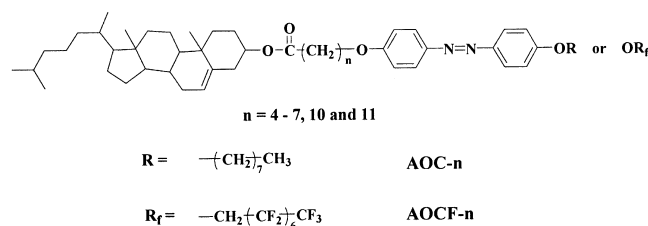
## 1. Introduction

Non-symmetric dimesogenic compounds [1–19] composed of two different mesogenic units are a relatively new class of LC compounds that still require much more study to understand their structure – property relationships. We have already reported a wide variety of polymorphism exhibited by the non-symmetric dimesogens [3–6, 10, 11, 15–19]. The possibility of the formation by those compounds of uncommon phases such as incommensurate phases, the blue phase and the twisted grain boundary (TGB) phases, is rather intriguing. Most of the non-symmetric dimesogens described earlier by ourselves contained a cholesterol moiety as an integral structural component, carrying several chiral centres. Yelamaggad *et al.* [13] also reported the LC properties of dimesogenic compounds in which one of the mesogenic units was the cholesterol moiety. Luckhurst *et al.* [9] reported LC properties of dimesogenic compounds consisting of a Schiff's base mesogen bearing a chiral terminal alkyl chain and the cyanobiphenyl mesogen. Many research groups have reported a wide variety of non-symmetric and symmetric dimesogenic compounds without chiral centres [1, 4, 9, 20].

The effect of fluorinating aromatic mesogenic cores and of polyfluorinated alkyl or alkoxy chains on the LC properties of monomeric compounds has also been the

subject of many studies [21–29]. Wide mesomorphic temperature ranges, low melt viscosities and ferroelectricity of fluorinated compounds (with or without chiral centres) appear to be the major reasons for many of these studies. The possible technological applications of these compounds are also considered to be very important.

Recently, we compared the LC properties, including the ferroelectric behaviour, of a series of non-symmetric dimesogenic compounds in which the cholesterol moiety is interlinked through the 6-oxyhexanoyl spacer to the second mesogen having either an octyloxy or perfluoroheptylmethoxy terminal chain [19]. In particular, it was observed that the perfluoro chain in ferroelectric compounds brought about a much shorter response time, 80  $\mu$ s, than those with the alkoxy chain, 400–500  $\mu$ s. In this study we compare the LC properties of two series of dimesogenic compounds on varying the length of the central spacer linking the two mesogenic units. The AOC-*n* series have an octyloxy chain while the AOCF-*n* series possess a perfluoroheptylmethoxy tail, '*n*' denoting the number of methylene groups in the spacer:



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We were particularly interested in investigating (i) the dependence of LC behaviour on the length and parity of the methylene spacer, (ii) the dependence of LC behaviour on the nature of the chains on the azobenzene mesogen and (iii) the induction of the formation of the chiral smectic phase, especially of the SmC\* phase, by the chiral centres in the cholesterol moiety.

## 2. Experimental

### 2.1. Synthesis

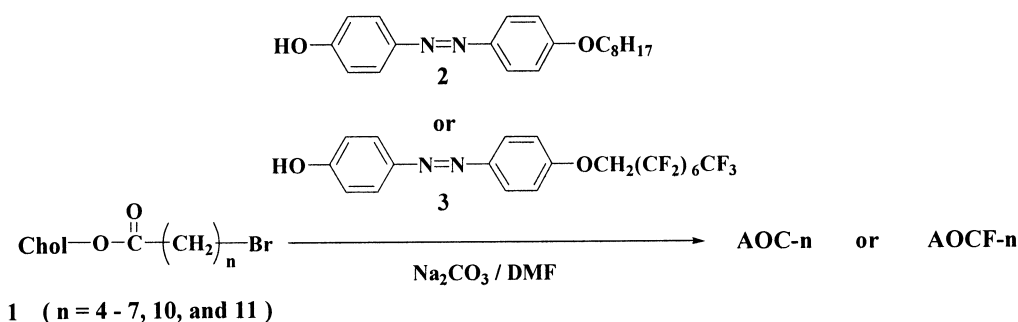
The AOC-*n* and AOCF-*n* series were prepared following the synthetic routes shown in schemes 1 and 2. The details of the synthetic procedures have already been reported earlier by ourselves [19] and others [30, 31] for similar compounds, and thus here we present only representative spectral and elemental analysis data for the new compounds and their intermediates.

As shown in scheme 1, the two series, AOC-*n* and AOCF-*n*, were prepared by reacting a cholesteryl  $\omega$ -bromoalkanoate **1** with 4-[(4-octyloxyphenylimino)methyl]phenol **2** or 4-[[4-(1H,1H-perfluorooctyloxy)phenylimino]methyl]phenol **3** in *N,N*-dimethylformamide in the presence of sodium carbonate. For preparative details and LC properties of AOC-5 and AOCF-5, see [19]. Details for the preparation of compound **2** can be

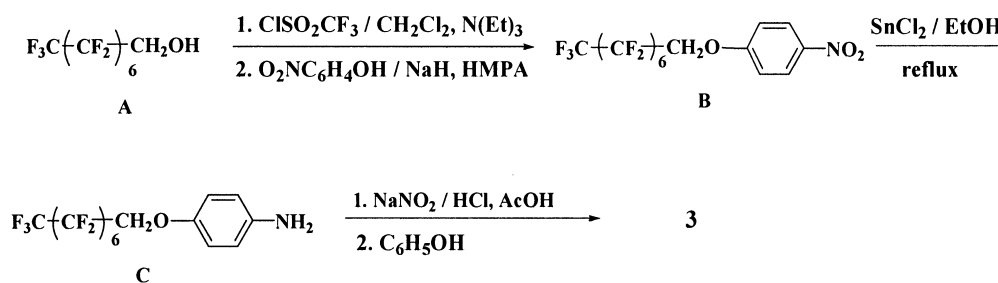
found in [32]. The route to compound **3** is shown in scheme 2[19]; it was prepared by the azo coupling reaction [30, 31] between phenol and 4-(1H, 1H-pentadecafluorooctyloxy)phenylamine (**C** in Scheme 2), itself obtained by reduction of the corresponding nitro compound (**B** in scheme 2). The nitro compound **B** was readily prepared by the nucleophilic substitution of pentadecafluorooctyl trifluoromethylsulphonate with sodium nitrophenolate.

**2.1.1. 4-[5-(Cholesteryloxycarbonyl)butyloxy]-4'-octyloxyazobenzene (AOC-4).** <sup>1</sup>H NMR ( $\delta$ , ppm):  $\delta$  0.67–2.04 (m, –CH–, –CH<sub>2</sub>– and –CH<sub>3</sub>), 2.23 (q, O–CH–CH<sub>2</sub>), 4.00 (t, CH<sub>2</sub>–O), 5.28 (d, C=CH), 6.96 (d, 4H, Ar), 7.84 (d, 4H, Ar). FTIR (KBr, cm<sup>-1</sup>): 2931 and 2852 (C–H stretching), 1728 (C=O stretching), 1602 and 1469 (aromatic C=C stretching), 1249 and 1172 (C–O stretching). Elemental analysis: calc. for C<sub>52</sub>H<sub>78</sub>N<sub>2</sub>O<sub>4</sub>, C 78.54, H 9.89, N 3.52, O 8.05; found, C 78.56, H 9.89, N 3.47%.

**2.1.2. 4-[5-(Cholesteryloxycarbonyl)butyloxy]-4'-perfluorooctyloxyazobenzene (AOCF-4).** <sup>1</sup>H NMR ( $\delta$ , ppm):  $\delta$  0.60–1.91 (m, –CH–, –CH<sub>2</sub>–, –CH<sub>3</sub>), 2.29 (m, –C=C–CH<sub>2</sub>–, –O–CO–CH<sub>2</sub>–), 4.03 (t, –CF<sub>2</sub>CH<sub>2</sub>O–), 4.55 (m, –OCH–), 5.38 (m, –C=CH–), 7.01 (d, 4H, Ar), 7.07 (d, 2H, Ar), 7.91 (t, 2H, Ar). FTIR (KBr,



Scheme 1. Synthesis of AOC-*n* and AOCF-*n*.



Scheme 2. Synthesis of 4-(1H, 1H-perfluorooctyloxy)-4'-hydroxyazobenzene, **3**.

$\text{cm}^{-1}$ ): 2934 and 2859 (aliphatic C–H stretching), 1730 (C=O stretching), 1600 (aromatic C=C stretching), 1238 and 1160 (C–O stretching). Elemental analysis: calc. for  $\text{C}_{52}\text{H}_{63}\text{F}_{15}\text{N}_2\text{O}_4$ , C 58.64, H 5.96, F 26.76, N 2.63, O 6.01; found, C 58.64, H 5.96, N 2.52%.

## 2.2. Identification and characterization

After purifying the intermediates and final products either by recrystallization from suitable solvents or by column chromatography on silica gel columns, their structures were verified by spectroscopic and elemental analyses. The FTIR and  $^1\text{H}$  NMR spectra of the compounds were recorded on a Bomem MB FTIR (Canada) spectrophotometer using solid KBr pellets or liquid films, and on a Bruker AM 300 Spectrometer (Germany) with TMS as internal standard, respectively.

The thermal phase transition temperatures were determined under  $\text{N}_2$  using a Mettler Toledo DSC 821e (Switzerland) differential scanning calorimeter. The heating and cooling rates were kept at  $5^\circ\text{C min}^{-1}$ . Indium was used as a reference metal for the calibration of temperature and enthalpy. The peak maximum or

minimum points on DSC thermograms were taken as the transition temperatures. The thermal properties and optical textures of the mesophases were observed on a polarizing microscope (Olympus BH-2, Japan) equipped with an automatically controlled (Mettler FP-90, Switzerland) hot stage (FP-82HT). X-ray diffractograms were obtained at varying temperatures using synchrotron radiation ( $1.542 \text{ \AA}$ ) of the 3C2 beam line at the Pohang Synchrotron Laboratory, Pohang, Korea. Identification of the  $\text{SmC}^*$  phases was performed by the observation of polarization switching using rubbed polyimide cells in addition to X-ray analysis.

## 3. Results and discussion

### 3.1. Phase transitions and mesophase stability

Figures 1(a) and 1(b) compare the DSC thermograms of the AOC-*n* and AOFC-*n* series. Tables 1 and 2 summarize the data for the thermal transitions and the nature of mesophases formed by the compounds. The DSC thermograms show that all the phase transitions occur reversibly or enantiotropically with the exception of AOC-6, AOC-10 and AOFC-10 which form  $\text{SmA}$  or

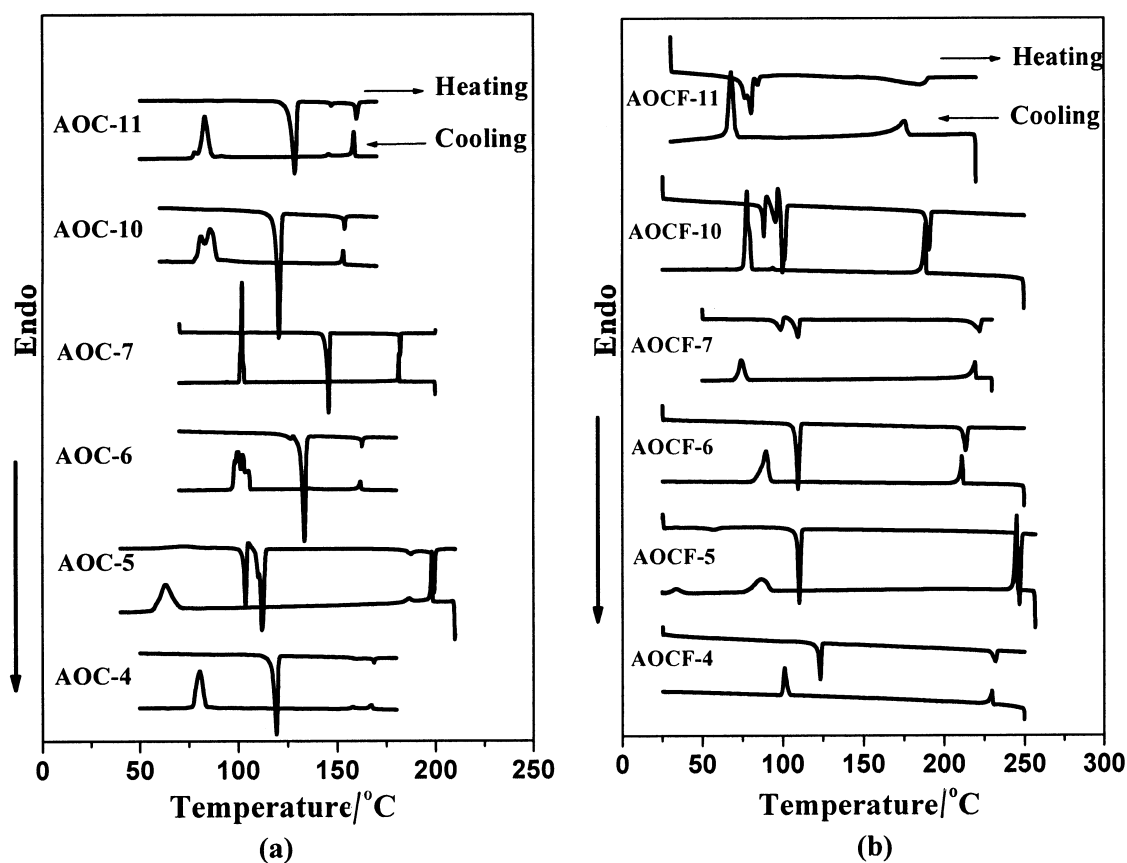


Figure 1. DSC thermograms of (a) AOC-*n*, (b) AOFC-*n*.

SmC\* phases monotropically. The SmC\*–SmA transition of AOC-5 was barely visible on the thermograms, but was confirmed by the changes in optical textures observed through the polarizing microscope. By contrast, the monotropic transition from the SmA to SmC\* phase is clearly discernible in the DSC thermogram of AOCF-10.

As for the melting transition temperatures ( $T_m$ ) of the two series, as shown in figures 2(a) and 2(b), the melting points (112–146°C) of the AOC- $n$  series are in general a little higher than those (80–124°C) of the AOCF- $n$  series and, in both series, a regular dependence

of  $T_m$  on the length ( $n$ ) and parity of the spacer is not apparent. The isotropization temperatures ( $T_i$ ), however, show a distinctive, so-called odd–even dependence on the number of methylene groups in the central spacer, although data points for  $n=8$  and 9 are missing. In both series the  $T_i$  values for compounds with the same spacer parity decreased with increasing  $n$ . It should be noted that odd values of  $n$  result in higher  $T_i$  values than even-numbered spacers, because the presence of the ester and ether groups between the two linked mesogenic units makes the apparently odd membered spacer actually contain an even number of

Table 1. Thermodynamic data for the phase transitions of AOC- $n$  series.

$n$	Transition temperature/°C (Enthalpy change/kJ mol <sup>-1</sup> )	$T_i$ (AOCF- $n$ ), $\Delta(T_i - T_k)$ /°C	
4	Cr 119.2 SmA 161.2 N* 167.6 I (55.4) (1.2) (2.0)	231.3	48.4
5 <sup>a</sup>	Cr 112.0 SmC* 135.7 SmA 187.5 N* 199.0 I (24.6) (0.04) (1.3) (5.4)	246.8	87.0
6	Cr 132.7 N* 162.6 I (41.7) (2.3) SmA ←	214.0	29.9
	135.9 (0.2)		
7	Cr 145.7 SmA 153.6 N* 180.9 I (36.7) (0.2) (5.9)	221.8	35.2
10	Cr 118.5 N* 153.7 I (45.2) (3.80) SmA ←	191.0	35.2
	121.0 (0.1)		
11	Cr 128.5 SmA 147.0 N* 160.0 I (48.8) (0.97) (6.99)	184.2	31.5

<sup>a</sup>Data taken from ref. [19].

Table 2. Thermodynamic data for the phase transitions of AOCF- $n$  series.

$n$	Transition temperature/°C (Enthalpy changes/kJ mol <sup>-1</sup> )	$T_i$ (AOC- $n$ ), $\Delta(T_i - T_k)$ /°C	
4	Cr 123.6 SmA 231.3 I (35.9) (13.9)	167.6	107.7
5 <sup>a</sup>	Cr 110.1 SmA 246.8 I (21.8) (17.6)	199.0	136.7
6	Cr 110.5 SmA 214.0 I (42.3) (13.2)	162.6	103.5
7	Cr 110.3 SmA 221.8 I (21.7) (14.7)	180.9	111.5
10	Cr 101.3 SmA 191.0 I (47.6) (15.7) SmC* ←	153.7	89.7
	95.4 (0.6)		
11	Cr 80.1 SmA 184.2 I (24.9) (12.6)	160.0	104.1

<sup>a</sup>Data taken from ref. [19].

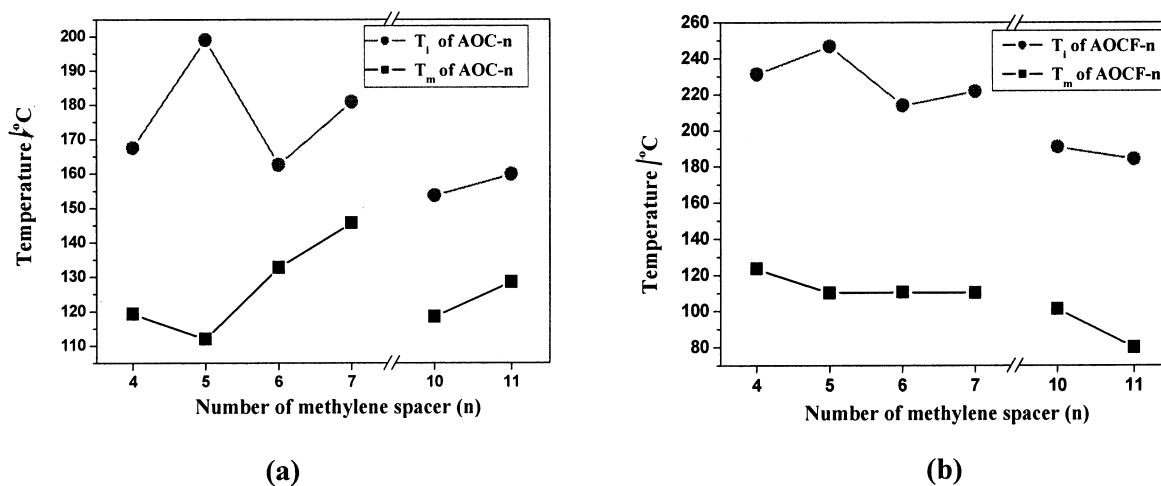


Figure 2. The dependence of  $T_m$  and  $T_i$  on the number of the methylene unit ( $n$ ) in (a) AOC- $n$  and (b) AOCF- $n$ .

atoms in the spacer and vice versa. In other words, in the present case, the compounds with odd values  $n$  exhibit higher  $T_i$ s than those with even values of  $n$ .

Similar behaviour has been observed in the series by ourselves [33]. Various molecular theories have been proposed to explain the alternation of the transition

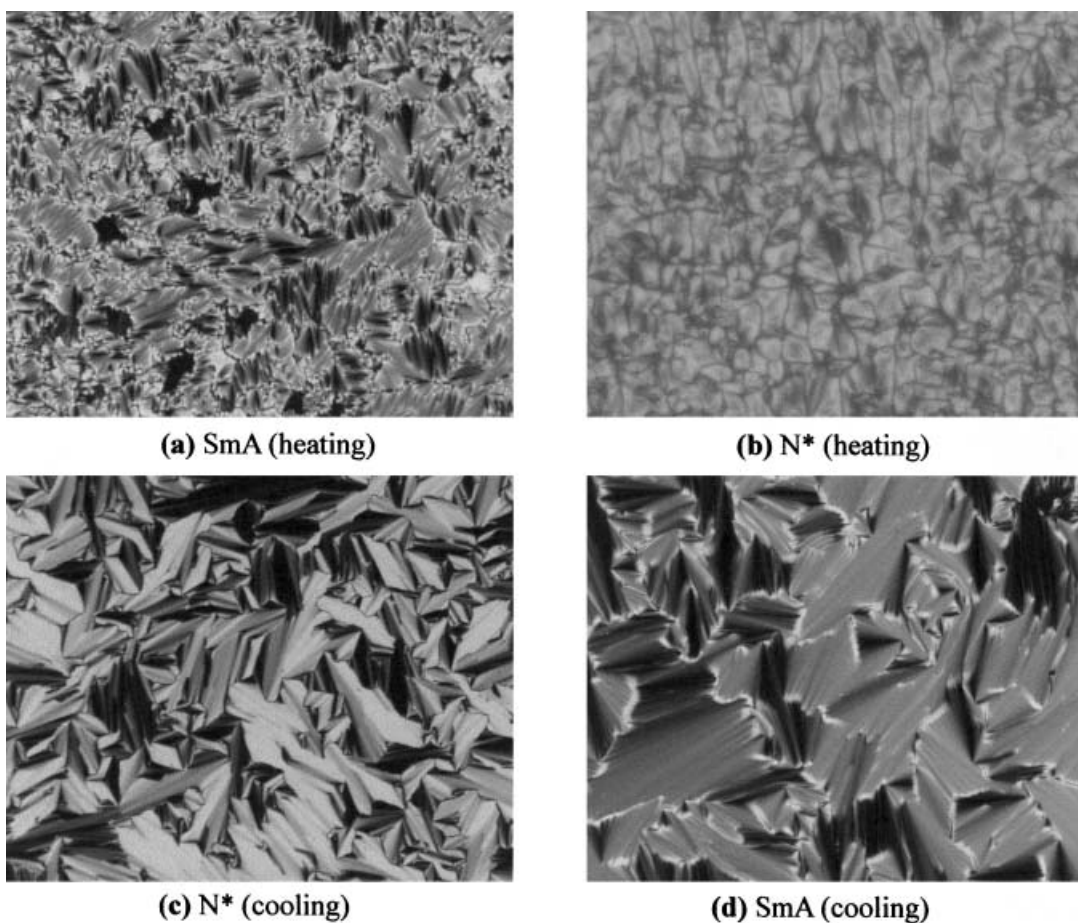


Figure 3. Photomicrographs of (a) AOC-11 taken at 141.0°C heating, (b) AOC-4 taken at 163.0°C heating, (c) AOC-11 taken at 156.7°C cooling and (d) AOC-11 taken at 144.8°C cooling (magnification 400×).

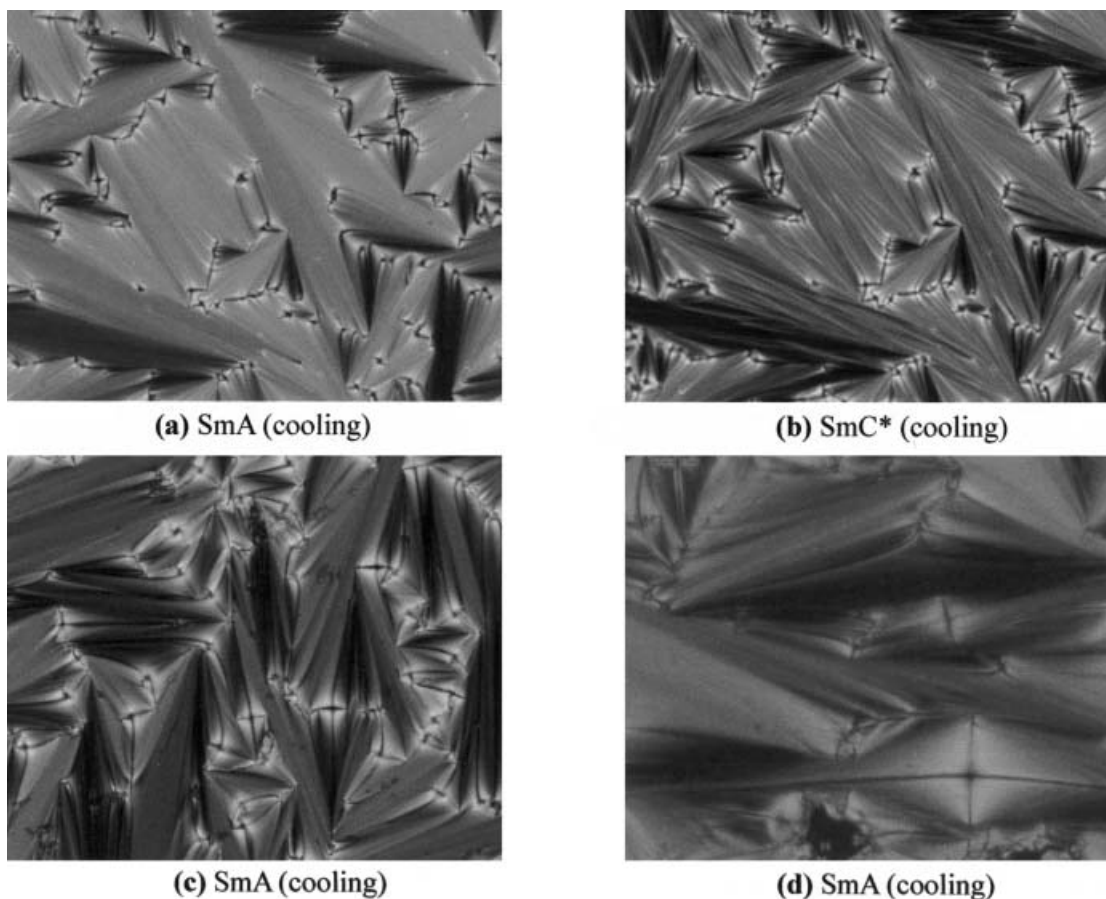


Figure 4. Photomicrographs of (a) AOCF-10 taken at 172.3°C (b) AOCF-10 taken at 87.9°C (c) AOCF-6 taken at 189.5°C and (d) AOCF-4 taken at 185.0°C on cooling (magnification 400×).

properties with the number of the methylene groups in the central spacers of liquid crystal dimers [34].

A scrutiny of the transition temperature data in tables 1 and 2 leads us to another important finding: the isotropization temperatures ( $T_i$ ) of the AOCF- $n$  series are far higher than those of the AOC- $n$  series. This, in turn, increases the mesophase temperature range of the former. The  $T_i$  values of the AOCF- $n$  series are higher by 24–51°C than those of the corresponding members of the AOC- $n$  series. The mesophase temperature range of the AOC- $n$  series is 30–87°C (for most of them only about 30°C), whereas the range for fluorinated series is 90–137°C, typically about 100°C. Thus, the introduction of fluorine atoms in the terminal alkoxy chain increases the thermal stability of the mesophases of the resulting compounds. These observations may be attributed to (i) the perfluorinated alkyl chain's higher tendency to reside in the all-*trans* conformation [35, 36] when compared with hydrogenated alkyl groups and (ii) their strong incompatibility with hydrogenated chains, which drives microphase

separation [37, 38]. These points will be further discussed later.

### 3.2. Mesophase structures

The nature of the mesophases formed by the present compounds was determined by DSC, polarizing microscopy and X-ray diffraction. In addition, the LC behaviour of AOC-5 and AOCF-5 reported earlier in detail by ourselves, [19] provides a reliable basis for the identification of mesophases exhibited by other members of the present series. The optical textures shown in figures 3 and 4 show typical focal-conic (SmA), focal-conic with dark homeotropic regions (SmA), broken focal-conic (SmC\*), and fan-shaped (N\*) textures. In order to clarify further the structure of smectic phases, we obtained X-ray diffractograms in the small angle region and representative examples are shown in figure 5. Comparison of the data in tables 1 and 2 reveals that the AOC- $n$  series form a greater variety of mesophases than the AOCF- $n$  series. The former exhibit

SmA, SmC\* and N\* phases, whereas the latter only the SmA phase with the one exception of AOCF-10 that forms the SmC\* phase monotropically. AOC-5 is unique in that it is the sole compound among the AOC-*n* members that shows a SmC\* phase. This peculiarity may be explained by the relatively low enthalpy of melting (24.6 kJ mol<sup>-1</sup>) of this compound when compared with the enthalpy of melting (37–55 kJ mol<sup>-1</sup>) of other compounds. The ferroelectric properties of AOC-5 were reported earlier by ourselves, [19]: its spontaneous polarization value was about P<sub>s</sub>=400 nC cm<sup>-2</sup> and response time of 250–600 μs, depending on temperature. All the AOC-*n* compounds exhibit the cholesteric (N\*) phase. By contrast the AOCF-*n* series form only the SmA phase with the exception of AOCF-10. None of them show the cholesteric (N\*) phase although they do contain a cholesterol moiety. In other words, the perfluoroalkoxy group totally suppresses the formation of the N\* phase, as observed earlier by ourselves [19] and others [27–29]. It is also known that fluorination suppresses the nematic character of nematogens [39]. Guittard *et al.* [21] published a review article several years ago about highly fluorinated thermotropic liquid crystals and discussed various aspects associated with fluorine substituents.

As mentioned already, perfluoroalkyl groups, due to strong lateral forces between them, exhibit incompatibility with both saturated and aromatic hydrocarbons; this, in turn, leads to microsegregation favouring smectic LC morphologies. Consequently, the formation of nematic and cholesteric phases is suppressed when the length of the perfluoroalkyl group is not too short.

Table 3 lists the layer spacings of the smectic phases estimated from the small angle X-ray diffractograms (e.g., figure 5). We note from these data that the layer spacings of AOCF-4 to AOCF-7 are slightly greater than their molecular lengths calculated using CS Chem 3D Pro (Cambridge Soft Co., U.S.A.) assuming a *trans*-conformation for all the CH<sub>2</sub> and CF<sub>2</sub> carbons in the central spacer and in the terminal chain. This indicates that the assumption is appropriate. In contrast, when the spacer length is increased as in AOCF-10 and AOCF-11, the layer spacings become a little shorter than the molecular length, most probably due to the presence of *gauche* conformations in some CH<sub>2</sub> group(s) in the spacer.

All the smectic layer spacings of the AOC-*n* series are smaller than the molecular lengths calculated for fully extended conformers. This suggests a higher possibility exists for the CH<sub>2</sub> groups in the spacers and in the terminal chains to adopt *gauche* conformations. In fact,

it is known that fluorinated alkyl groups have a tendency to reside in more extended conformations than alkyl groups, due to a greater energy difference (9.1 kJ mol<sup>-1</sup>) between the *trans*- and *gauche*-conformations [36]. The energy difference for hydrogenated alkyl groups is 3.4 kJ mol<sup>-1</sup>.

Figure 6 shows how abruptly the layer spacing of the SmA phase of AOCF-10 decreases from 53.6 to 50.5 Å as it undergoes the phase transition from SmA to SmC\*. The tilt angle of the SmC\* phase is estimated to be about 19.5°. The ferroelectric properties of this compound are presently under investigation, although they may not differ significantly from those of AOCF-5 reported earlier by ourselves [19].

#### 4. Conclusion

We successfully prepared two new series of non-symmetric dimesogenic compounds consisting of cholesterol and azobenzene-based mesogens interconnected by ω-oxyalkanoyl spacers of varying length. The first series (AOC-*n*) contain an octyloxy chain on the azobenzene mesogen, while the second series (AOCF-*n*) has a perfluoroheptylmethyloxy chain. The two series show distinctively different mesomorphic behaviour. The AOC-*n* series exhibit SmC\*, SmA and N\* phases depending on the length of the central spacer. In contrast, the perfluoroalkyl chain in the AOCF-*n* series suppresses the formation of SmC\* and N\* phases. Instead, it favours the formation of the SmA phase, which is attributed to its strong incompatibility with the hydrogenated segments.

In addition, the perfluoroalkyl chain tends to improve the thermal stability of the mesophase,

Table 3. Layer spacings in the SmA and SmC\* phases for the AOC-*n* and AOCF-*n* series.

Compound	Layer spacing/Å		Estimated molecular length/Å
	SmA phase	SmC* phase	
AOC-4	45.3		47.0
AOC-5 <sup>a</sup>	47.8	41.3	48.1
AOC-6	47.8		49.1
AOC-7	49.1		50.3
AOC-10	50.8 and 24.3		53.9
AOC-11	52.0 and 26.0		55.3
AOCF-4	48.7		47.8
AOCF-5 <sup>a</sup>	49.9		48.3
AOCF-6	51.0		49.2
AOCF-7	52.0		51.2
AOCF-10	53.6	50.5	54.3
AOCF-11	55.2		55.7

<sup>a</sup>Data taken from ref. [19].



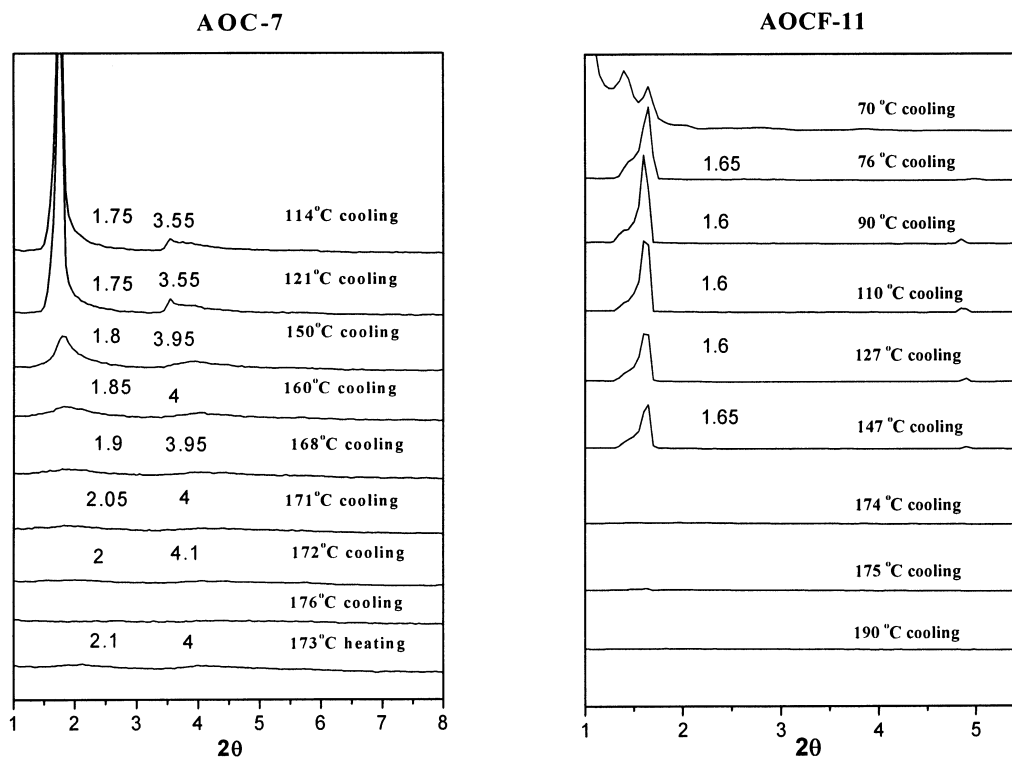


Figure 5. Small angle X-ray diffractogram of AOC-7, AOCF-11.

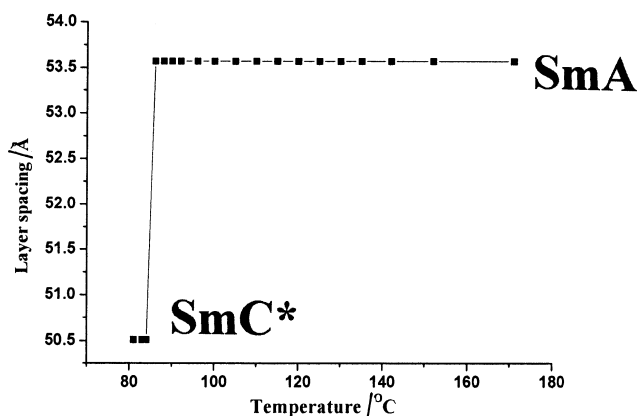


Figure 6. Temperature dependence of layer spacing of AOCF-10.

increasing the mesophase temperature range, which is believed to be due to strong interactions among the perfluoroalkyl tails. AOCF-10 is an exception in that it forms the SmC\* phase monotropically.

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